Serial No. 09/831.915

DOCKET NO: 208608US0PCT

## IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

THOMAS DANIEL, ET AL. : EXAMINER: METZMAIER, D. S.

SERIAL NO: 09/831,915 :

FILED: MAY 25, 2001 : GROUP ART UNIT: 1712

FOR: HYDROGELS CAPABLE OF ABSORBING AQUEOUS FLUIDS

## DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Dr. Volker Braig who deposes and states:

- 1. That I am a graduate of RWTH Aachen and received a Ph.D. degree in chemistry in the year 2001.
- 2. That I have been employed by BASF SE (formerly BASF Aktiengesellschaft), since 2001 as a chemist in the field of polymer chemistry.
- 3. That the following experiments were carried out by me or under my direct supervision and control.

The comparative samples were prepared in accordance with WO 97/46195 (Procter and Gamble) (see page 6, 4<sup>th</sup> paragraph and the examples). In Example 1 of WO '195, AGM, zeolite and silica were used in a ratio of 67:50:50. In the present experiment, these compounds were in the same ratio.

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WO 97/46195 is silent about the structure of the AGM that was used. It is noted that AGM is a superabsober as is SAP. AGM is a sodium polyacrylate (SAP). As shown by "Modern Superabsorbent Polymer Technology" (attached and incorporated herein by reference) commercially available superabsorbents are based on lightly crosslinked partially neutralized polymers of acrylic acid. The crosslinking makes the polyacrylate insoluble. The attachment from Fresenius refers in item 4 to AGM as superabsorber which is polyacrylate.

Further, WO 97/46195 is silent whether sodium silicate is used as solid or as aqueous solution. Therefore, sodium silicate was used in both forms.

SAP, sodium water glass and zeolites in powder form were used for sample [1]. In sample [2], the starting soda water glass was in liquid form. The superabsorber SAP was mixed with additives (water glass and zeolites) under various conditions described below.

#### **SAMPLE DESIGNATIONS:**

Sample [1] was also designated as EDG-0007-17-A.

Sample [2] was also designated as EDG-0007-17-B.

<sup>&</sup>lt;sup>1</sup> As shown in US 2009/0118689, paragraph [0016], AGM as used in WO 97/46195 (Procter and Gamble) is a superabsorber just as SAP. Paragraph [0016] states that [0016]"Absorbent polymer material," "absorbent gelling material," "AGM," "superabsorbent," and "superabsorbent material" are used herein interchangeably and refer to cross linked polymeric materials that can absorb at least 5 times their weight of an aqueous 0.9% saline solution as measured using the Centrifuge Retention Capacity test (Edana 441.2-01).

## PREPARATION OF SAMPLE 1 (EDG-0007-17-A)

6.7 g of SAP from Dow Chemical Corp., 5.0 g zeolite (zeolite H-MFI-90, Süd-Chemie AG, München) and 5.0 g solid sodium silicate (Brilesil®C335 from PQ Europe GmbH) were given into a 100 ml square bottle and homogenized for 20 min. at 32 rpm using a dry-blend mixer (Turbula (R), Willy A. Bachofen AG - Maschinenfabrik, Switzerland). The resulting sample was in the form of a free flowing powder. See the attached photograph.

# PREPARATION OF SAMPLE 2 (EDG-0007-17-B)

6.7 g of SAP from Dow Chemical Corp. and 5.0 g zeolite (zeolite H-MFI-90, Süd-Chemie AG, München) were weighed into a mixing container. The container was closed with a cover that had an opening for syringes and a flow breaker. Then, the mixing containing was set on top of the Waring blender (Blender 8012, Model 34BL99, Waring Laboratory, U.S.) and briefly homogenized at level 1.

Then, 14.08 g of 35.5 wt.% aqueous solution of sodium silicate (equal to 5g of sodium silicate powder) were added to the Waring blender using a one-way syringe making sure that the syringe was completely filled. The aqueous solution of sodium silicate was added while the blender was operated at level 4 (out of 7 possible levels). After the aqueous solution of sodium silicate was added, the blender was shut off. Any crusting on the walls of the mixing container was removed with a spatula and the Waring blender was briefly turned on.

The sodium silicate solution was obtained from Woellner GmbH & Co. KG, Ludwigshafen.

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There was no drying after the mixing with sodium silicate as the resulting product was a free-flowing powder. See the attached photograph. The powder was filled into a 100 ml square bottle.

The so obtained samples were then used by Dr. Essig to prepare samples for SEM measurements.

- 4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.
  - 5. Further deponent saith not.

Signature  $\frac{Mag}{Date} = \frac{10^{44}}{2010}$